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To: • Mr. J. M. Madures date: January 7, 1975
From: • E. L. Watts
Subject: • A Product Evaluation of DIET versus Westab ET

INTRODUCTION AND OBJECTIVES

The purpose of this project was to determine the physical, chemical, and subjective differences between Westab expanded tobacco (ET) and dry ice expanded tobacco (DIET) by evaluating each of the products at the 100% level and at various substitution levels in a Marlboro blend.

The three specific objectives of this evaluation were as follows:

- A. To determine the filling power effects of the reordering of the exit tower product of both processes.
- B. To determine the filling power effects of thermo-vac treatment of DIET versus ET.
- C. To define physical, chemical, and subjective differences between DIET and ET substituted at various levels in a Marlboro type blend.

The bright filler used as the control for both processes was part of a normal 20,000 pound line cut for Westab expansion. Six thousand pounds of filler (carefully sampled so as to be representative of the 20,000 pound line) was sent to R&D for expansion by the DIET process. The remaining fourteen thousand pounds were expanded at Westab by the ammonia-CO₂ process.

RESULTS AND DISCUSSION

Evaluation of 100% DIET versus ET

Filling Power Evaluation

Samples of the control and the expanded product were taken at the following locations:

Control - conveyor leading to impregnator

Expanded product - after the tower, before reordering unit

Reordered product - after reordering unit, before air leg

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Control samples and P/S reordered samples were submitted for cylinder volume determination on an "as is" basis. Samples taken out of the tower before the reordering unit were equilibrated on a "blow-box" (75 F, 60% R.N., 30 ft/min. air flow) for approximately eighteen hours before cylinder volume determinations were made. Results of the cylinder volume testing are summarized below and in Table I.

<u>Sample Location</u>	<u>C.V. at 11% O.V.</u>	<u>C.V. at 11% O.V.</u>	<u>Δ CV</u> <u>(ET-DIET)</u>
	<u>Westab ET</u>	<u>DIET</u>	
Equilibrated - Out of Tower	68.5	68.8	-0.3
"As Is" - Out of P/S	68.6	63.5	+5.1
Equilibrated - Out of P/S	65.3	66.5	-1.2

Data obtained on equilibrated samples out of the tower and after the Proctor Schwarz (P/S) reordering unit show no significant differences between the filling power of the DIET versus ET. "As is" cylinder volume results on P/S reordered material indicate that the Westab ET was higher in filling power than the DIET product by ~7%. It should be noted at this time that no effort has been made to optimize conditions in the P/S reordering unit used at R&D. This may have had some effect on the lower C.V. result for the DIET.

Chemical Evaluation

There were significant chemical differences between the ET and DIET products as shown in Table II. Both products showed significant reduction in alkaloid and total reducing sugar levels, however, the decrease in reducing sugar content was much more severe in the Westab ET product. Due to impregnation with ammonia for the ET process, the product was characteristically higher in amino sugars, total nitrogen content, percent total volatile bases, and soluble ammonia. Significant losses of propylene glycol and glycerin were also noticed, especially in the case of the DIET material.

Sieve Evaluation

Samples were taken at the boxing site for sieve fraction determinations to "flag" any major differences in particle size distribution of the final products. There was a slight difference in the "long" and "medium" fractions, with Westab having a higher percentage of "longs". Results are shown in Table III.

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Thermo Vac Treatment of DIET versus ET

In order to determine the effects of thermo treatment on the filling power of DIET versus ET, ten hogsheads of each product were sent to the Commerce Road facility for processing. Ten samples for C.V. determinations were pulled from each hogshead as it was being filled to be used as a "before thermo" control. After exposure to the formal beetle kill cycle for ET and ES, the twenty hogsheads were returned to R&D to obtain C.V. samples "after thermo." An individual record of C.V. before and after thermo was kept for all twenty hogsheads. A summary of this testing is listed in Table IV, where the results represent averages of the ten hogsheads of each product. Samples were run both on an "as is" basis and on an equilibrated basis. In both cases Westab ET and DIET showed a decrease in C.V. through the thermo process.

	<u>Westab ET</u>	<u>DIET</u>
% Loss in filling power "As Is" Basis	5.6	4.0
% Loss in filling power Equilibrated Basis	3.6	2.2

After thermo, Westab ET was higher in filling power than the DIET, and as was found in the testing of out of the tower and after P/S samples, the magnitude of difference was less for the equilibrated samples than for the "as is" samples.

A composite sample of DIET and ET to be used in making blends of various levels of the two products was prepared by combining portions of each of the ten treated hogsheads.

Evaluation of DIET versus ET at Various Substitution Levels in a Marlboro Type Blend

Marlboro-type blends were prepared containing 4, 10, 20, and 33% ET and DIET for the purpose of making cigarettes for firmness determinations. A sample containing no expanded tobacco was used as a control for the testing. In preparing the blends with increasing percentages of ET or DIET, only the percentage bright component of the blend was reduced.

Cylinder Volume Data--Before Cigarette Making

Samples of each of the final blends were pulled throughout the run for cylinder volume determinations. Hand blends were also prepared in which an exact amount of ET or DIET was weighed into the

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the blend to yield the same percentages of expanded material targeted for in the larger blends. A comparison of C.V. results obtained for the hand blends versus results obtained for the larger scale blends is made in Table V. The C.V. values for the hand blends act as a guide to the filling power increase expected due to the increase in ET or DIET percentages in the blend. The agreement between the corrected C.V. values for hand blends and the larger scale blends indicates that the large scale blends appear to contain the proper levels of expanded material. Target cigarette making weights were then calculated from the corrected C.V. values obtained for the large scale blends.

One additional comment should be made about the hand blend C.V. data in Table V: the cylinder volume values for the blends containing ET tended to be higher than those obtained on the DIET blends.

Cigarette Firmness Data

Cigarettes made from the control blend, all of the ET and DIET blends, and the two 100% expanded materials were evaluated for firmness. Results of this testing are summarized in Table VI and plotted in Graph I. The weight reductions determined for each blend due to increased percentages of ET or DIET were calculated using the control weight and the weights of the test cigarettes at equal firmness. From these weight reductions and the target percentages of ET or DIET in the blend, an average weight reduction per 1% ET or DIET added was calculated.

Overall, the firmness data indicated there was no significant difference in the filling power of ET and DIET as shown by the average weight reductions per 1% material added. These values are listed below:

<u>Expanded Tobacco Level</u>	<u>Wt. Reduction/ 1% ET</u>	<u>Wt. Reduction/ 1% DIET</u>
4%	1.47	1.62
10%	0.73	0.90
20%	0.79	0.72
33%	0.63	0.52
100%	0.43	0.40
Average Wt. Reductions/1% Added	0.81	0.83

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Cylinder Volume Data - Ripped Cigarettes

Cylinder volume data on ripped cigarettes (Table VI) showed no significant differences in filling power at the 4% and 10% levels, however at the 20%, 33%, and 100% levels the blends containing the Westab ET tested slightly higher in filling power than the corresponding DIET blends. The significance of these differences is uncertain.

Chemical Data on Cigarettes

Cigarettes at the target making weight of each of the blends and 100% materials were submitted to Analytical for smoking, chemical, and picking analysis. These results are listed in Table VII. As the percentages of DIET and ET in the blends increased, the FTC Tar, nicotine, and puff counts decreased. There were no appreciable differences in these values between the cigarettes containing DIET and those containing ET, except at the 33% and 100% levels. At these high levels of substitution the DIET cigarettes delivered less "tar".

Chemical analysis of the blends showed a reduction in alkaloid content for both the DIET and ET blends while a reduction in total reducing sugars was noticed only in ET blends as the substitution level increased. Picking results indicated that the percentages of total reconstituted material and ES were consistent throughout all the blends, although they tended to be slightly below target. This consistency lends a certain amount of confidence that the changes in filling power observed were due to the different target levels of ET or DIET substituted in the blend. Picking values for DIET and ET were always lower than the target levels.

Subjective Evaluation of Cigarettes

It was intended to include a subjective evaluation (RP³ Test) of the DIET versus ET at the 4 and 10% levels, however the flavor panel detected a slight "off taste" in the 10% DIET cigarette. By smoking the 100% DIET cigarette versus the 100% ET cigarette it was determined that the "off taste" was not due to the DIET, but due to some problem in the remaining portion of the blend. A request has been submitted to remake the four blends and remake the cigarettes for the subjective evaluation. The results of this testing will be reported at a later date.

CONCLUSIONS

From the evaluation of 100% DIET versus 100% ET, it was found that there was no significant difference between the two products from a

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filling power standpoint when samples out of the tower and out of the Proctor Schwarz (P/S) were equilibrated prior to cylinder volume testing. However, cylinder volume results on material from the P/S showed Westab ET to be higher in filling power by ~7% if the samples were run on an "as is" basis. The reason for differences between "as is" and equilibrated data is still uncertain.

Chemically, the two products were different in total reducing sugar content, and final propylene glycol (PG) and glycerin levels. The DIET product showed greater losses of PG and glycerin through the tower. Due to impregnation with ammonia, the ET was characteristically higher in total nitrogen, total volatile base, and soluble ammonia levels. Both products showed considerable alkaloid reduction over the control bright filler.

No significant differences were observed in sieve results obtained on each of the final products.

Thermo treatment of the DIET and ET yielded a reduction in cylinder volume of 4.0% and 5.6% respectively on samples run on an "as is" basis. The difference between "as is" data and equilibrated data was also observed in this test. Equilibrated results showed reductions of 2.2% and 3.6% for DIET and ET respectively. Cylinder results obtained in this testing tended to show ET to be slightly higher in filling power than DIET both before and after thermo treatment.

Firmness test results obtained on cigarettes containing blends of 4, 10, 20, and 33% DIET and ET and each of the 100% products showed that, on the average, an equal weight reduction of approximately 0.8% per 1% expanded material added could be taken for both products. Cylinder volume results on ripped cigarettes showed no significant differences in filling power at the 4 and 10% levels, however, at the 20, 33, and 100% levels the blends containing the Westab ET tested slightly higher in filling power than the corresponding DIET blends.

Smoking and chemical analyses showed reductions in FTC tar, nicotine, puff count, and total alkaloids as the level of expanded material in the blend increased. There were no significant differences between the DIET and ET cigarettes except at the 33% and 100% levels where the DIET cigarettes delivered less "tar". Total reducing sugar content of the blends decreased as the level of ET increased, whereas no such trend was observed in the DIET blends.

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Subjective testing of the 4% and 10% levels of DIET versus ET has not been completed at this time. Results of this testing will be reported at a later date.

RECOMMENDATIONS

It is recommended a series of "mini tests" be run to monitor chemical and filling power differences between Westab ET and DIET. Comparisons will be made on both products out of the tower and after the reordering unit. Arrangements have been made with Westab personnel to pull samples from their process when notified by R&D to do so.

The results obtained from the series of "mini tests" should give some indication as to whether some of the differences observed in this large scale test are significant.

/mc

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TABLE I

CYLINDER VOLUME DATA - 100% DIET VS. 100% ET

	Westab ET	DIET	Δ CV (Westab ET-DIET)
<u>Control (As Is)</u>			
CV (as is)	35.6	36.2	
OV (as is)	11.54	11.36	
CV at 11% O.V.	37.0	37.6	
*C.F.	-2.6	-4.0	
<u>Tower Product (Equilibrated)</u>			
"As Is" O.V.	3.24	1.55	
CV (Equil.)	61.9	70.4	
OV (Equil.)	11.72	10.79	
CV at 11% O.V.	68.5	68.8	-0.3
*C.F.	-9.1	-7.7	
<u>P/S Reordered Product (As Is)</u>			
CV (as is)	68.4	70.1	
OV (as is)	11.02	10.33	
CV at 11% O.V.	68.6	63.5	+5.1
*C.F.	-11.1	-9.8	
<u>P/S Reordered Product (Equilibrated)</u>			
CV (Equil.)	66.2	67.3	
OV (Equil.)	10.9	10.9	
CV at 11% O.V.	65.3	66.5	-1.2
*C.F.	-9.1	-7.7	

*C.F. = correction factor determined from computer analysis of CV and OV data.

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TABLE II

CHEMICAL ANALYSIS ON CONTROL VS. 100% ET VS. 100% DIET

Analysis ¹	Westab Control	DIET Control	Avg. Control	Westab ET	DIET
OV(after grinding),%	10.5	10.3	10.4	10.2	9.6
Alkaloids, %	2.60	2.48	2.54	1.63	1.52
Total Reducing Sugars, %	14.2	14.0	14.1	4.0	10.8
Sucrose, %	0.5	0.5	0.5	0.6	0.9
Fructose, %	6.7	6.8	6.8	0.4	5.8
Glucose, %	4.3	4.2	4.3	N.D. ²	1.7
Amino Sugars, %	0.32	0.33	0.33	0.52	0.30
Total Nitrogen, %	2.21	2.33	2.27	4.19	2.36
TVB, %	0.38	0.40	0.39	1.36	0.25
Ash, %	12.2	12.7	12.4	12.8	13.0
Pet. Ether Ext., %	6.3	6.6	6.5	7.3	8.1
SoL. NH ₃ , %	<0.1	<0.1	<0.1	0.82	N.D. ²
PG, %	1.9	1.9	1.9	0.8	0.3
Gly., %	1.4	1.4	1.4	1.2	0.9
CO ₂ , %	-	-	-	0.2	0.1

¹15 samples of control, 100% ET and 100% DIET submitted for analysis in duplicate²N.D. = Not Detected³All results on Dry Weight Basis

Diff. Alk., Sugar, PG, Gly. (reductions in both products)

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TABLE III

SIEVE ANALYSIS - SAMPLES TAKEN AT BOXING SITE

	<u>Westab ET R&D Results</u>	<u>Westab ET Westab Plant Results</u>	<u>DIET R&D Results</u>
% Longs	34.88	(35.22)	29.82
% Mediums	51.56	(52.88)	57.18
% Shorts	10.95	(8.85)	10.91
% Smalls	1.24	(1.58)	1.26
% Fines	1.21	(1.50)	0.83
O.V., %	10.5	(10.8)	10.4

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TABLE IV
CYLINDER VOLUME DATA - HOGSHEADS - BEFORE AND AFTER THERMO TREATMENT

	<u>Westab ET</u>	<u>DIET</u>	<u>AC.V. (Westab ET-DIET)</u>
<u>Hogsheads-Before Thermo (As Is)</u>			
CV (as is)	75.7	73.8	
OV (as is)	10.6	10.1	
CV at 11.0% OV	71.3	65.0	+6.3
C.F.	-11.1	-9.8	
<u>Hogsheads-After Thermo (As Is)</u>			
CV (as is)	71.7	72.2	
OV (as is)	10.6	10.0	
CV at 11.0% OV	67.3	62.4	+4.9
C.F.	-11.1	-9.8	
<u>ΔCV Through Thermo Process</u>	-4.0	-2.6	
<u>Hogsheads-Before Thermo (Equilibrated)</u>			
CV (Equil.)	72.7	66.4	
OV (Equil.)	10.9	11.2	
CV at 11.0% OV	71.8	67.9	+3.9
C.F.	-9.1	-7.7	
<u>Hogsheads-After Thermo (Equilibrated)</u>			
CV (Equil.)	70.1	65.6	
OV (Equil.)	10.9	11.1	
CV at 11.0% OV	69.2	66.4	+2.8
C.F.	-9.1	-7.7	
<u>ΔCV Through Thermo Process</u>	-2.6	-1.5	

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TABLE V

CYLINDER VOLUME DATA - COMPARISON OF LARGE SCALE BLENDS TO HANDBLENDED SAMPLES

<u>SAMPLE I.D.</u>	<u>LARGE SCALE BLEND SAMPLES</u>			<u>HANDBLEND SAMPLES</u>		
	<u>C.V.</u>	<u>O.V.</u>	<u>CORRECTED C.V.*</u> <u>AT 12.5% O.V.</u>	<u>C.V.</u>	<u>O.V.</u>	<u>CORRECTED C.V.*</u> <u>AT 12.5% O.V.</u>
Control - 0% ET	38.0	12.82	39.0	35.8	13.46	38.7
4% Westab ET	36.2	13.45	39.0	37.1	13.47	40.1
4% DIET	39.2	12.65	39.3	37.3	13.21	39.4
10% Westab ET	38.2	13.25	40.5	38.9	13.46	41.8
10% DIET	38.9	12.93	40.2	39.8	12.75	40.6
20% Westab ET	41.4	12.64	41.8	41.4	13.10	43.2
20% DIET	42.3	12.42	42.1	40.1	13.05	41.8
33% Westab ET	46.7	12.34	46.2	44.7	13.10	46.5
33% DIET	46.6	12.12	45.5	44.2	12.75	45.0

* Cylinder volumes corrected to 12.5% O.V. level using a correction factor of 3.0 cc/10 gms/1% O.V. change.

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TABLE VI
COMPACIMETRIC FIRMNESS DATA

SAMPLE I.D.	CIGT. TOB. WT.	CIGARETTE FIRMNESS	EQUIL. O.V.	TOB. WT. AT EQUAL FIRM.	% WEIGHT REDUCTION	C.V.-RIPPED CIGARETTES	EQUIL. O.V.
D4BFS	0.850	36.8	13.3				
Control 0% ET	0.817	39.2	13.3	0.818	-	34.2	13.4
	0.767	42.2	13.4				
D4BFK	0.820	36.1	12.9				
4% Westab ET	0.766	39.2	12.9	0.770	5.86	36.2	13.0
	0.719	42.0	12.8				
D4BFO	0.816	36.0	12.9				
4% DIET	0.781	38.0	12.9	0.765	6.47	36.2	12.8
	0.735	40.8	12.8				
D4BFL	0.807	36.2	13.1				
10% Westab ET	0.754	39.8	13.1	0.758	7.33	39.0	12.8
	0.709	41.9	12.8				
D4BFP	0.801	35.7	12.8				
10% DIET	0.759	38.4	12.8	0.744	9.04	38.5	12.7
	0.720	42.1	12.8				
D4BFM	0.782	33.1	12.9				
20% Westab ET	0.730	36.6	13.1	0.688	15.89	40.6	12.6
	0.689	38.9	13.0				
D4BFQ	0.766	35.0	13.1				
20% DIET	0.713	37.9	13.0	0.700	14.42	39.3	12.7
	0.672	40.7	12.9				
D4BFN	0.720	34.4	12.9				
33% Westab ET	0.670	37.6	12.7	0.648	20.78	43.8	12.6
	0.620	40.8	12.7				
D4BFR	0.712	36.8	13.0				
33% DIET	0.659	40.2	13.0	0.678	17.11	42.1	12.8
	0.610	43.2	12.8				
D4BFT	0.513	33.7	11.7				
100% Westab ET	0.468	38.6	11.7	0.464	43.27	61.4	11.8
	0.419	42.3	11.8				
D4BFU	0.518	35.6	11.5				
100% DIET	0.469	40.8	11.5	0.491	39.37	60.2	11.3
	0.418	44.3	11.3				

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TABLE VII
SMOKING AND CHEMICAL ANALYSIS OF CIGARETTES

	D4875 CONTRAL-OS ET	D4876 45 WESTAR ET	D4870 45 DIET	D4871 10X WESTAR ET	D4877 10X DIET	D4878 20X WESTAR ET	D4879 20X DIET	D4880 33X WESTAR ET	D4881 33X DIET	D4882 100X WESTAR ET	D4883 100X DIET
Smoke											
Butt Length, mm	28	28	28	28	28	28	28	28	28	28	28
FTC Tar mg/cigt.	17.1	16.9	17.6	17.4	17.9	17.6	16.6	16.3	16.4	15.7	15.5
Nicotine, mg/cigt.	1.20	1.11	1.14	1.09	1.12	1.02	1.05	0.89	0.88	0.84	0.85
Puffs/cigt.	8.9	9.2	9.2	8.7	8.8	8.5	8.4	8.0	7.6	6.1	5.9
Cigarette											
Total H ₂ O, in. of H ₂ O	4.6	4.6	4.7	4.3	5.0	5.3	5.0	5.3	4.9	5.4	5.8
Mod H ₂ O, in. of H ₂ O	1.9	1.9	2.1	1.6	2.3	2.6	2.4	2.5	2.1	2.7	3.0
Static Burn Time, min.	7.6	7.8	8.2	7.7	7.7	7.8	7.8	7.5	7.0	6.9	6.3
Length, mm	84.3	84.6	84.3	84.6	84.4	84.3	84.6	84.5	84.3	84.3	84.3
Circumference, mm	25.1	25.0	25.1	25.0	25.1	25.1	25.0	25.1	25.3	25.1	25.0
% Cool Removal	14	18	20	18	19	12	7	16	32	6	12
Paper											
Porosity, sec.	21	18	19	23	21	21	22	28	21	21	23
Filler											
Total Alkaloids, %	1.70	1.64	1.65	1.58	1.67	1.59	1.56	1.74	1.42	1.43	1.48
Total Reducing Sugars, %	6.0	6.4	6.8	6.3	5.9	4.4	6.2	3.8	6.4	3.1	9.0
PG, %	1.5	1.2	1.1	3.2	1.2	1.2	1.1	1.2	1.2	0.8	0.5
Gly, %	1.9	1.4	1.5	1.5	1.5	1.7	1.7	1.8	1.9	2.2	1.7
Tab. Vol., gms.	0.797	0.776	0.804	0.750	0.757	0.748	0.721	0.674	0.657	0.471	0.465
Mod Density, gm/cc	0.257	0.245	0.253	0.237	0.238	0.235	0.228	0.212	0.204	0.148	0.147
Reconstituted tab., % (Target=16.65)	15.51	15.03	16.19	17.65	16.20	16.64	15.16	16.04	16.43	None	None
E.T., %	None	3.85	2.46	7.63	7.71	12.95	16.58	27.58	21.87	100	100
E.S., % (Target=6.758)	6.85	5.01	6.16	6.22	5.95	6.31	7.68	5.99	6.19	None	None
Open Volatiles, %	12.0	12.2	12.1	12.1	12.1	12.0	11.9	11.8	11.7	11.8	10.1

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